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Removal of Copper From Molten Ferrous Scrap: Results of Laboratory Investigations

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CONTENTS

TABLES

UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	m	meter
cm	centimeter	min	minute
g	gram	pct	percent
in	inch	vol pct	volume percent
kg	kilogram	wt pct	weight percent
L	liter		

REMOVAL OF COPPER FROM MOLTEN FERROUS SCRAP: RESULTS OF LABORATORY INVESTIGATIONS

By L. L. Oden[1] and G. W. Elger[2]

ABSTRACT

The Bureau of Mines has studied a number of processes to remove Cu from molten steel scrap with the objective of determining if any of the laboratory methods studied warranted in depth research investigations. Experimental approaches included oxidizing Cu from steel containing other metals that form stable mixed oxides with Cu (Al, Cr, Fe, Mn, Mo, and V), injecting the same metals as oxides to simplify the mechanism of complex oxide formation, partitioning Cu into alkali or alkaline earth silicate slags or oxide-moderated sulfides, and coremoving Cu and S by injecting active compounds of Ca and Mg. The most promising tests, producing nearly 0.3 pct Cu in the slag, used Ar injection of K_2O-moderated Na_2S-FeS below the surface of molten steel containing about 0.6 pct Cu and protected by a CaO-SiO_2 slag; however, in depth research investigations currently are not felt to be warranted.

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INTRODUCTION

The importance of ferrous scrap as a resource in the domestic iron and steel industry has increased significantly during the last two decades owing to the growth of steel production in electric arc furnaces. In 1984, approximately 55 million tons of scrap was consumed in domestic steel mills, of which 56 pct was used in electric furnaces, 28 pct in basic oxygen furnaces, 7 pct in open hearth furnaces, and 7 pct in blast furnaces (1).[3] Scrap presently is the most important source of iron, with more of the iron used in steelmaking coming from scrap than from the ore-blast furnace-basic oxygen furnace route (1).

Steelmaking technology has changed considerably within the same timeframe, with substantial shift of steel production from large integrated producers to regional minimills. These highly productive and energy-efficient minimills use electric furnaces, scrap charges, and continuous casting technology to provide nearly a 4-to-1 primary energy advantage over integrated producers (2, p. 17). The integrated steel producers, on the other hand, have more flexibility in that they can add pig iron, hot metal from blast furnaces, direct-reduced iron, or home scrap as a diluent for tramp elements in purchased scrap used in the charge mixture. However, it is not normal practice for electric furnace steelmakers to use hot metal or pig iron as a diluent.

The principal tramp elements of concern to producers of plain-carbon and low-alloy steels are Cr, Cu, Mo, Ni, and Sn (1, 3-5), of which Cu presents the most problems. In a 1978 survey of the steel industry made by Luria Brothers and Co., Inc., Cleveland, OH, 71 pct of the 87 responding steel companies reported that Cu contamination in scrap caused them the most problems in steel processing (6). Cu and Sn cause hot shortness in steel by forming a liquid layer in the grain boundaries under the oxide surface. Inland Steel Co. recently reported that purchased scrap usually contains 0.20 to 0.27 pct Cu, while most steel products must contain 0.15 pct or less Cu (7). In addition, Oregon Steel Mills, Portland, OR, reported that purchased scrap contains between 0.2 and 0.35 pct Cu (8). Cascade Rolling Mills, Inc., McMinnville, OR, has encountered problems with off-grade steel heats containing Cu levels of 0.5 pct and higher (9). Luria Brothers reports that the Cu problem in scrap has worsened since the firm's last survey, published in 1978 (10). Nucor Corp. reported that copper levels in purchased scrap have increased steadily during the past few years and presently are approaching 0.40 pct (11).

Several factors have promoted increased Cu levels in ferrous scrap. The current low prices for ferrous scrap and red metal have lessened the economic incentive to handpick the Cu in the form of electric motors, wiring, etc., from scrap such as that derived from obsolete automobiles (7). The increased usage of high-strength, low-alloy (HSLA) steels, which may contain up to 1.25 pct Cu, 1.80 pct Cr, and 5.25 pct Ni, eventually affects scrap quality. Another significant source that lowers the quality of ferrous scrap is the carbon steels that are purposely alloyed with 0.2 to 0.4 pct Cu, such as U.S. Steel COR-TEN.

Another factor that impacts on scrap quality is that many minimills have switched to higher quality steel products in order to compete favorably with imported steel. As a result, more individual steel mills are placing stricter specifications on purchased scrap used to produce high-quality steel products (12-14). This practice may eventually lead to shortages of premium grades of ferrous scrap and result in higher scrap prices.

The scrap processors currently use hand sorting, mechanical shredding, and magnetic separation techniques to separate the nonferrous metals, glass, and other non-iron-bearing materials from ferrous scrap. About 200 shredders process approximately 60 pct of the discarded automobiles (4, p. K-2) to produce a product

[3]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

containing 0.2 pct Cu, 0.02 pct Sn, 0.16 pct Cr, and 0.04 pct Ni as the principal tramp elements (4, p. G-4). The various grades of processed scrap are summarized by Swager (4, pp. D1-D21).

Another physical separation technique, known as cryogenic processing, is employed to a limited extent in Europe to produce a premium grade of scrap (2, p. 40; 15-16), but in the authors' opinion, this practice appears to be too expensive for U.S. use.

Several physical and chemical techniques have been investigated on a laboratory and pilot scale to remove Cu from ferrous scrap. Evaporation of Cu from molten scrap under vacuum has been studied by several investigators (17-19). Thermal treatment and selective oxidation of Cu in scrap mixtures were investigated by Bureau researchers (20-24). Treatment techniques using either sulfide slags (25-30) or molten Pb baths (29, 31) effectively lowered the Cu levels in molten, C-saturated iron, but several problems remain to be solved before the processes could be applied to molten steel.

Sulfide slags, which have been investigated extensively, are not effective in removing Cu from molten ferrous scrap having low C contents (28). The activity of both Cu and S in molten iron is enhanced significantly as the C level is increased to saturation (29). In addition, a large slag volume is required as Cu exhibits a relatively high activity in fused Na_2S. In the case of a Pb bath to remove Cu from molten scrap, Langenberg reported the high vaporization rate of Pb from the bath is a major problem (29).

An alternate approach to physical-chemical treatment of ferrous scrap is neutralization of the effects of tramp elements such as Cu in steel. Copeland and coworkers at the Bureau of Mines found that additions of Al, B, Ni, and Si, to steel melts reduced or eliminated the hot shortness problem, depending on the Cu content (32, pp. 1-2; 33, p. 3). The effects of the steel additives on the structures and properties of the castings have not been established.

To be most useful, a process to remove tramp elements from ferrous scrap should be applicable to steel as well as cast irons, should not unduly extend the total refining time or create serious offgas or effluent problems, and must be effectively inexpensive. The ideal process would be pyrometallurgical in nature and would consist of simple partitioning of Cu into a slag that could be recycled following Cu recovery. In the present work, numerous pyrometallurgical processes were considered, both conceptually and experimentally, to remove Cu from molten steel scrap. The objective of this limited and somewhat qualitative test work was to evaluate some concepts that had not appeared previously in the literature. Positive results were needed to justify a future comprehensive study.

EXPERIMENTAL WORK

Scrap charges comprising 25- to 30-kg ingots of premelted shredded automobile scrap were melted within an alumina crucible in a 45-kg-capacity induction furnace. Materials to be evaluated were injected as fine particles below the surface of the molten scrap through a nominal 3/8-in (0.9525-cm-ID) Fe lance using air, O_2-enriched air, or Ar carrier gas. Injection required about 10 min, during which time 2 to 4 m of Fe lance (1.2 to 2.4 kg) was melted into the steel. Following injection of the material, a sample of slag was recovered on a cold steel rod, and a spoon sample of metal was cast into a 2.54-in-diam by 2.54-cm-high alumina mold. The slag sample was pulverized, and any occluded metal was removed by a hand magnet prior to analysis by atomic absorption. The metal sample was ground flat on the bottom for X-ray emission analysis and drilled on the side to provide material for C and S analyses by the combustion method. Most of the difference between the initial and final Cu concentrations in the steel is the result of dilution of the steel by the Fe lance.

RESULTS

A number of pyrometallurgical processes were evaluated to remove Cu from molten steel. The initial effort was designed to exploit the stability of Cu compounds under oxidizing conditions. These tests depend upon the formation of oxide species in regions of high O_2 potential obtained by sparging with air or O_2-enriched air. Numerous complex oxides containing Cu, as listed in table 1, were investigated using two techniques:

1. Copper and the other metal in the complex oxide were dissolved in the steel, and air or air-50 vol pct O_2 was sparged into the steel to effect oxidation. Slag and metal were removed immediately for analysis before general equilibrium between slag and metal was attained, because the complex oxides are thermodynamically unstable in the presence of Fe.

2. Copper was dissolved in the steel, and the metal oxide of interest was injected under the surface of the melt with air, air-50 pct O_2, or Ar. Slag and metal again were removed immediately for analysis.

The complex oxides and their melting or decomposition temperatures are listed in table 1, and the results of the experiments are given in table 2. The formation of complex oxides containing Cu by gas injection of an existing oxide is mechanistically simpler than the multibody reaction involving the oxidation of two metals in solution, which may explain the observation that injection of the oxides was generally much more effective than in situ oxidation. The oxides V_2O_5 and Cr_2O_3 were more effective than Al_2O_3 and MnO_2. Note that the higher S content of the steel for the oxide injection tests, where comparisons can be made (Cr_2O_3, NiO, Fe_2O_3, and V_2O_5), resulted in less copper reporting to the slag. Copper removal is better for the low-S steel; however, for the method to be economically viable, the Cu level in the slag must be increased 20 to 80 times above the results obtained thus far, even in the best cases.

Numerous alkali silicates and complex alkali-alkaline earth silicates and aluminates were studied by injecting the finely divided material below the surface of the molten steel with air,

TABLE 1. - Complex oxides containing copper

Chemical formula	Melting or decomposition temp in air, °C	Remarks
$Cu_2O \cdot Al_2O_3$....	1,240	Decomposes at 1,026° C to $CuO \cdot Al_2O_3$ in air.
$CuO \cdot Al_2O_3$.....	1,200	
$CuO \cdot 2CaO$.....	1,035	
$Cu_2O \cdot CoO_2$....	1,070	Decomposes at 915° C to liquid + Cu_2O in air.
$Cu_2O \cdot Cr_2O_3$....	>1,500	Decomposes at 890° C to $CuO \cdot Cr_2O_3$ + CuO in air.
$CuO \cdot Cr_2O_3$.....	1,100	
$CuO \cdot Fe_2O_3$.....	~1,600	
$Cu_xMn_{3-x}O_4$....	>1,400	Cubic (spinel) solid solution.
$CuO \cdot MnO$.....	1,155	Decomposes at 1,060° C to $CuO \cdot Mn_2O_3$ + CuO in air.
$Cu_{1.7}Mn_{1.3}O_3$..	1,110	Decomposes at 910° C to spinel + CuO in air.
$3CuO.2MoO_3$....	880	Decomposes at 812° C to $CuO \cdot MoO_3$ + $2CuO \cdot MoO_3$ in air.
$5CuO \cdot V_2O_5$.....	816	
$3CuO \cdot V_2O_5$.....	780	
$2CuO \cdot V_2O_5$.....	~780	
$CuO \cdot V_2O_5$.....	650	
$Cu_2O \cdot P_2O_5$.....	1,245	

TABLE 2. - Copper removal by in situ oxidation and by injection of oxides

Additive		Vol of gas injected, L			Cu in steel, wt pct[1]		Initial slag present[2]	Cu in final slag, wt pct
Type	Amount, g	Air	Air-50 vol pct O_2	Argon	Initial	Final		
Metals:								
Al.....	52	76	NAp	NAp	0.48	0.46	No	0.0026
Co.....	113	305	NAp	NAp	.52	.44	No	.022
Cr.....	178	178	NAp	NAp	.47	.44	No	.0013
	178	NAp	156	NAp	.46	.38	No	.067
Mn.....	212	414	NAp	NAp	.54	.47	No	.014
Mo.....	183	404	NAp	NAp	.53	.43	No	.015
Ni.....	113	206	NAp	NAp	.52	.47	No	.020
V.....	197	312	NAp	NAp	.51	.46	No	.022
Oxides:								
γ-Al_2O_3..	157	103	NAp	NAp	.46	.48	No	.022
CaO.....	221	NAp	192	NAp	.64	.63	No	.016
Cr_2O_3....	293	NAp	208	NAp	.74	.66	No	.045
	300	NAp	200	NAp	[3].57	.49	Yes	.026
	300	NAp	NAp	200	[3].59	.57	Yes	.020
Fe_2O_3....	300	NAp	198	NAp	.65	.63	No	.037
	300	NAp	100	NAp	[3].52	.50	Yes	.025
	300	NAp	NAp	122	[3].55	.52	Yes	.021
MnO_2.....	418	NAp	164	NAp	.70	.65	No	.027
MoO_2.....	278	NAp	199	NAp	.73	.69	No	.031
NiO.....	180	NAp	129	NAp	.70	.65	No	.042
	300	NAp	NAp	95	[3].56	.52	Yes	.027
	300	NAp	200	NAp	[3].52	.46	Yes	.023
TiO_2.....	300	NAp	180	NAp	.65	.61	No	.023
V_2O_5.....	360	NAp	160	NAp	.71	.66	No	.076
	300	NAp	[4]117	NAp	[3].56	.50	Yes	.031

NAp Not applicable.

[1]25 to 30 kg of steel contained 0.02 to 0.05 wt pct S and 1 to 1.5 wt pct C before test unless otherwise noted. The difference between initial and final Cu contents is largely the result of dilution by the Fe injection lance.

[2]Initial slag composition: 80 g CaO and 60 g SiO_2.

[3]Steel contained 0.3 to 0.4 wt pct S.

[4]100 pct O_2.

air-50-vol pct O_2, or Ar. Justification for studying these materials lies in the stability of $CuSiO_3$ and the propensity for Cu to substitute in complex alkali metal silicates. A case in point is the mineral lititidionite ($NaKCuSi_4O_{10}$), which is a stable liquid phase at the melting point of Fe (34). The Fe analog of these silicates may also be stable, and in fact may be more stable than the Cu-containing compound. Hence, a local environment of high alkali or alkaline earth oxide concentration may be advantageous, as provided by injecting the silicates and aluminates below the surface of the molten metal. The results, given in table 3, are not encouraging, although some interesting observations are apparent. For example, based on slag analyses, $2K_2O \cdot SiO_2$ is 3 times more effective than $2Na_2O \cdot SiO_2$ and 10 times more effective than $2Li_2O \cdot SiO_2$.

The effectiveness of sulfides to remove Cu from C-saturated iron has been recently reaffirmed by Liu and Jeffes (35), who studied Na_2S slags containing FeS and CaO. They concluded that 70 wt pct Na_2S-30 wt pct FeS was the best

TABLE 3. - Copper removal by injection of silicates and aluminates

Compound injected		Vol of gas injected, L			Cu in steel, wt pct[1]		Initial slag present[2]	Cu in final slag, wt pct
Type	Amount, g	Air	Air–50 vol pct O_2	Argon	Initial	Final		
[3]$Na_2O \cdot K_2O \cdot SiO_2$	414	107	NAp	NAp	0.49	0.46	No	0.017
Portland cement	440	115	NAp	NAp	.49	.48	No	.014
[3]$2Li_2O \cdot SiO_2$	284	NAp	137	NAp	.49	.47	No	.006
[3]$2Na_2O \cdot SiO_2$	280	NAp	117	NAp	.65	.60	No	.019
[3]$2K_2O \cdot SiO_2$	372	NAp	203	NAp	.64	.61	No	.058
	300	NAp	150	NAp	.54	.48	Yes	.037
	300	NAp	NAp	104	.55	.54	Yes	.052
$KAlSi_3O_8$ (potassium feldspar)	300	NAp	179	NAp	.65	.60	No	.038
	300	NAp	200	NAp	[4].53	.49	Yes	.027
	300	NAp	NAp	132	[4].56	.53	Yes	.029
$Mg_3Si_4O_{10}(OH)_2$ (talc)	300	NAp	171	NAp	.64	.59	No	.014
[5]$CaO \cdot Al_2O_3$	274	NAp	130	NAp	.70	.66	No	.024
[6]$Na_2O \cdot Al_2O_3$	300	NAp	159	NAp	.64	.61	No	.023
Amorphous SiO	300	NAp	140	NAp	.65	.61	No	.025
$LiAlSi_2O_6$ (spodumene)	300	NAp	180	NAp	.65	.61	No	.023
$(NaK)AlSiO_4$ (nepheline syenite)	300	NAp	167	NAp	.64	.61	No	.024

NAp Not applicable.

[1]25 to 30 kg of steel contained 0.02 to 0.05 wt pct S and 1 to 1.5 wt pct C before test unless noted otherwise. The difference between initial and final Cu content is largely due to dilution by the Fe injection lance.

[2]Initial slag composition: 80 g CaO and 60 g SiO_2.

[3]Prefused and ground mixture of carbonate(s) and silica sand.

[4]Steel contained 0.3 to 0.4 wt pct S.

[5]Prefused and ground mixture of lime and γ-alumina.

[6]Prefused and ground mixture of NaOH and γ-alumina.

TABLE 4. - Copper removal by injection of oxide-moderated sulfides

(Weight percent unless otherwise specified)

Compound injected[1]	Vol of Ar injected, L	Cu in steel[2]		S in steel[2]		Cu in slag[3]	S in slag[3]
		I	F	I	F		
Na_2S-FeS-CaO	127	0.56	0.52	0.041	0.31	0.07	4.47
Na_2S-FeS-K_2O	82	.56	.51	NA	NA	.27	NA
Na_2S-FeS-MgO	100	.60	.57	NA	NA	.12	NA
Na_2S-FeS-Y_2O_3	103	.62	.57	NA	NA	.08	NA
Na_2S-FeS-Ce_2O_3	105	.61	.58	NA	NA	.10	NA
Na_2S-FeS-MnO_2	117	.62	.58	NA	NA	.18	NA
Na_2S-FeS-BaO	103	.83	.78	NA	NA	.18	NA

NA Not analyzed. I Initial. F Final.

[1]189 g Na_2S, 81 g FeS, and 30 g remaining constituent.

[2]25 to 30 kg of steel contained 0.02 to 0.05 wt pct S and 1 to 1.5 wt pct C before test. The difference between the initial and final Cu contents is largely due to dilution by the Fe injection lance.

[3]Initial slag composition: 80 g CaO and 60 g SiO_2.

composition and that the addition of CaO improved the transfer of Cu to the slag while impeding the transfer of S to the metal. The present study extended their work to evaluate other metal oxides where the metal either forms very refractory sulfides (Ca, Ce, Mg, Y) or forms strong bonds with S but forms a sulfide with a lower melting point. The C content of the iron in the present study was greater than 1 pct, but significantly less than C saturation. The cover slag for the test series, a slightly basic CaO-SiO_2, was present to retard the reversion of Cu.

The test results, given in table 4, are the most encouraging to date, with K_2O producing 0.27 pct Cu in the slag and both the MnO_2- and BaO-moderated slags producing 0.18 pct Cu in the cover slag. However, even in the best case, a 10- to 30-fold increase in Cu concentration in the cover slag is needed for a viable process.

Injection of CaC_2 and Fe-Si-Mg was designed to exploit the stability of the intermetallic compounds Ca_4Cu, $CaCu_5$, and $MgCu_2$. The latter is a very stable phase melting congruently at 815° C. The CaO-SiO_2 cover slag was modified with K_2CO_3 or with K_2SO_4 in separate tests to evaluate the effects of K and S in the cover slag on the reversion of Cu to the metal phase.

The test results, given in table 5, indicate a negative effect for K in the cover slag and a definite positive effect for S in the cover slag. The last two tests in table 5 were designed to evaluate the coremoval of Cu and S from iron using CaC_2 injection. Both tests indicate the effectiveness of CaC_2 to desulfurize the metal and indicate an improvement in Cu removal. However, in all cases the Cu concentration in the slag is over 100 times too small for a viable process.

TABLE 5. - Copper removal by injection of CaC_2 and Fe-Si-Mg

(Weight percent unless otherwise specified)

Compound injected		Vol of Ar injected, L	Slag Composition	Cu in steel[1]		C in steel[1]		S in steel[1]		Cu in final slag	S in final slag
Type	Amount, g			I	F	I	F	I	F		
CaC_2.....	320	70	[2]$CaO-SiO_2$.....	0.76	0.71	1.73	1.65	0.039	0.035	0.014	NA
CaC_2.....	320	51	[2]$CaO-SiO_2-K_2CO_3$.	.71	.69	1.65	1.59	.035	.032	.013	NA
CaC_2.....	320	50	[2]$CaO-SiO_2-K_2SO_4$.	.69	.65	1.59	1.51	.032	.066	.029	1.60
[3]Fe-Si-Mg.....	300	75	[2]$CaO-SiO_2$.....	.65	.62	1.51	1.43	.066	.065	.008	NA
[3]Fe-Si-Mg.....	300	84	[2]$CaO-SiO_2-K_2CO_3$	.62	.61	1.43	1.38	.065	.047	.002	NA
[3]Fe-Si-Mg.....	300	88	[2]$CaO-SiO_2-K_2SO_4$	.61	.58	1.38	1.31	.047	.110	.011	1.08
CaC_2.....	220	118	[4]SiO_2.....	.76	.73	1.77	1.69	.036	.019	.037	1.30
CaC_2.....	294	108	[4]$SiO_2-K_2SO_4$	.73	.69	1.69	1.67	.019	.016	.027	1.12
CaC_2.....	295	90	[4]SiO_2.....	.69	.66	1.68	1.70	.34	.15	.036	2.28
CaC_2.....	293	98	[4]$SiO_2-K_2SO_4$....	.66	.63	1.70	1.64	.15	.18	.042	4.40

NA Not analyzed. I Initial. F Final.

[1]25 to 30 kg of steel. The difference between the initial and final Cu contents is largely due to dilution by the Fe injection lance.

[2]240 g CaO, 180 g SiO_2, and 80 g remaining constituent, if any.

[3]8 wt pct Mg, 32 wt pct Si, 48 wt pct Fe.

[4]200 g SiO_2 and 90 g remaining constituent, if any.

SUMMARY AND CONCLUSIONS

Numerous pyrometallurgical processes were considered both conceptually and experimentally to remove Cu from molten steel scrap. Over 60 semiquantitative tests were conducted in searching for a positive indication of Cu removal. The resulting information, if any of the tests had been successful, would have formed the basis for future research. The most successful test produced nearly 0.3 pct Cu in a $CaO-SiO_2$ slag using Ar injection of K_2O-moderated Na_2S-FeS below the surface of the molten steel. However, in the authors' opinion, a viable process must provide at least 5 pct Cu in the slag, a level that seems unattainable by the methods employed in this test work.

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